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Preparation of aniline having chlorine atom and perhalogenated aliphatic group with fluorine atom, used as intermediates for e.g. pharmaceuticals, by contacting N-aryl carbamoyl fluoride with chlorinating agent in hydrofluoric medium

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Addnl. Data: SAINT JALMES L, SCHANEN V

B(10-B4A, 11-C1) C(10-B4A, 11-C1) .2

NOVELTY

Preparation of an aniline (I) having both a chlorine atom and a perhalogenated aliphatic group with at least one fluorine atom, on the aromatic ring, comprises contacting, at at least 50°C, a N-aryl carbamoyl fluoride with a chlorinating agent in a hydrofluoric medium capable of exchanging the chlorine and the fluorine on the benzyl position or on a carbon carrying a chalcogen.

DETAILED DESCRIPTION

An INDEPENDENT CLAIM is included for the preparation of an N-phenylpyrazole dichlorinated on the ortho-ortho'-position of the pyrazole and having an electro-attractor on the para position, which includes the stage of preparing (I) as above.

USE

Used for preparation of intermediates for pharmaceutical and agrochemical products, especially intermediates for the preparation of Fipronil.

ADVANTAGE

The process is easier to carry out, and produces less saline waste material, than known methods.

EXAMPLE

A reaction mixture from the fluorination of 4-trichloromethylphenyl isocyanate, containing 1 mole of carbamoyl 4-trifluoromethyl phenyl fluoride and 5.5 moles of hydrofluoric acid, was treated in a closed autoclave, with 2.2 molar equivalents of chlorine. The mixture was heated to 90-110°C for 2-3 hours, after which analysis showed that most of the aromatic compounds present had a carbamoyl fluoride and/or isocyanate group dichlorinated on the

aromatic ring, together with traces of 2,6-dichloro-4-trifluoromethyl aniline. The mixture was cooled and neutralized, and the solvent evaporated to give an oil (225 g) most of which was 2,6-dichloro-4-trifluoromethyl aniline.

TECHNOLOGY FOCUS

Organic Chemistry - Preferred Process: Chlorination is effected by bubbling chlorine into the mixture at atmospheric pressure or above and at a temperature of 70-120°C. The mixture is then rapidly cooled and hydrofluoric acid eliminated under reduced pressure. (16pp520DwgNo.0/0)

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